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भारतीय मानक

पेट्रोलियम एवं प्राकृतिक गैस उद्योग — द्रव हाइड्रोकार्बन के पाईपलाईन परिवहन में प्रयुक्त दबी अथवा निमग्न पाईपलाईन हेतु बाह्य लेप

भाग 1 पालीलेफिन लेप (3 लेयर पी ई और 3 लेयर पी पी)

Indian Standard

PETROLEUM AND NATURAL GAS INDUSTRIES — EXTERNAL COATINGS FOR BURRIED OR SUBMERGED PIPELINES USED IN PIPELINE TRANSPORTATION OF GAS AND LIQUID HYDROCARBONS

PART 1 POLYOLEFIN COATINGS (3 LAYER PE AND 3 LAYER PP)

ICS 25.220.99; 75.200

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 10

FOREWORD

This Indian Standard (Part 1) was adopted by the Bureau of Indian Standards, after the draft finalized by the Corrosion Protection and Finishes Sectional Committee had been approved by the Metallurgical Engineering Division Council.

Corrosion is the physio-chemical interaction between a metal and its environment that results in changes in the properties of the metal, and which may lead to significant impairment of the function of the metal. Corrosion affects every sector of industry as well as infrastructure. The avoidance and control of corrosion is, therefore, of the utmost importance.

While formulating this standard International practices have been duly considered along with specific Indian scenario.

This standard specifies requirements of plant applied external three layer extruded polyethylene and polypropylene based coatings for corrosion protection of welded and seamless steel pipes for pipeline transportation of gas and liquid hydrocarbons in the petroleum and natural gas industries.

This standard has been issued in two parts. The other part in the series is:

Part 2 Fusion bonded epoxy coating

The composition of the Committee responsible for the formulation of this standard is given in Annex M.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PETROLEUM AND NATURAL GAS INDUSTRIES — EXTERNAL COATINGS FOR BURRIED OR SUBMERGED PIPELINES USED IN PIPELINE TRANSPORTATION OF GAS AND LIQUID HYDROCARBONS

PART 1 POLYOLEFIN COATINGS (3 LAYER PE AND 3 LAYER PP)

1 SCOPE

This standard (Part 1) specifies requirements of plant applied external three layer extruded polyethylene and polypropylene based coatings for corrosion protection of welded and seamless steel pipes for pipeline transportation of gas and liquid hydrocarbons in the petroleum and natural gas industries. The applied coating fulfilling the requirements of this part of standard is considered suitable for the service, handling, storage, construction and operation conditions described in **9**.

NOTE — Coated pipes in accordance with this part of standard are considered suitable to further protection by means of cathodic protection.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

Standard	*Title*
IS 101 (Part 1/Sec 7) : 1987	Methods of sampling and test for paints, varnishes and related products: Part 1 Tests on liquid paints (general and physical) film formation, Section 7 Mass per 10 litres (*third revision*)
IS 101 (Part 3/Sec 2) : 1989	Methods of sampling and test for paints, varnishes and related products: Part 3 Tests on paint film formation, Section 2 Film thickness
ASTM D-1693 : 2005	Standard test method for environmental stress-cracking of ethylene plastics
IS 1872-2 : 1997	Plastics — Polyethylene (PE) moulding and extrusion materials: Part 2 Preparation of test specimens and determination of properties
IS 1978 : 1982	Line pipe (*second revision*)
ISO 4892-2 : 1994	Plastics — Methods of exposure to laboratory light sources — Part 2 : Xenon-arc sources
ISO 8501 (all parts)	Preparation of steel substrates before application of paints and related products — Visual assessment of surface cleanliness
ISO 8502-3 : 1992	Preparation of steel substrates before application of paints and related products — Tests for assessment of surface cleanliness — Part 3: Assessment of dust on steel surfaces prepared for painting (pressure sensitive tape method)
ISO 8503 (all parts)	Preparation of steel substrates before application of paints and related products — Surface roughness characteristics of blast-cleaned steel substrates
IS/ISO 9001 : 2000	Quality management systems — Fundamentals and vocabulary (*second revision*)
ISO 10474 : 1991	Steel and steel products — Inspection documents
IS 10951 : 2002	Polypropylene materials for moulding and extrusion (*first revision*)
ISO 11124 (all parts)	Preparation of steel substrates before application of paints and related products —

Standard	*Title*
	Specification for metallic blast-cleaning abrasives
ISO 11126 (all parts)	Preparation of steel substrates before application of paints and related products — Specification for non-metallic blast-cleaning abrasives
ISO 11127-6 : 1993	Preparation of steel substrates before application of paints and related products — Teat methods for non-metallic blast-cleaning abrasives — Part 6 : Determination of water soluble contaminants by conductivity measurement
ISO 11357-1 : 1999	Plastics — Differential scanning calorimetric (DSC) — Part 1 : General principles
ISO 11357-2 : 1999	Plastics — Differential scanning calorimetric (DSC) — Part 2 : Determination of glass transition temperature
ISO 11420 : 1996	Plastic pipes and fittings — Method of test for carbon black dispersion in polyethylene pipes and fittings
IS 13360 (Part 3/Sec 1) : 1995 ISO 1183 : 1987	Plastics — Method of testing: Part 3 Physical and dimensional properties, Section 1 Determination of density and relative density of non-cellular plastics
IS 13360 (Part 4/Sec 1) : 1995 ISO 1133 : 1997	Plastics — Method of testing: Part 4 Rhelogical properties, Section 1 Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics
IS 13360 (Part 5/Sec 2) : 1996 ISO 527-2 : 1993	Plastics — Method of testing: Part 5 Mechanical properties, Section 2 Determination of tensile properties — Test conditions for moulding and extrusion plastics
IS 13360 (Part 5/Sec 3) : 1999 ISO 527-3 : 1995	Plastics — Method of testing: Part 5 Mechanical properties, Section 3 Determination of tensile properties — Test conditions for films and sheets
IS 13360 (Part 5/Sec 5) : 1999 ISO 179 : 1993	Plastics — Method of testing: Part 5 Mechanical properties, Section 5 Determination of Charpy impact strength
IS 13360 (Part 5/Sec 11) : 1999	Plastics — Method of testing: Part 5 Mechanical properties, Section 11 Determination of indentation hardness of plastics by means of durometer (shore hardness)
IS 13360 (Part 6/Sec 1) : 1999 ISO 306 : 1994	Plastics — Method of testing: Part 6 Thermal properties, Section 1 Determination of vicat softening temperature (VST) of thermoplastic materials (*first revision*)
ISO 13623 : 2000	Petroleum and natural gas industries — Pipeline transportation systems
IS 14001 : 2004 ISO 14001 : 2004	Environmental management systems — Requirements with guidance for use (*first revision*)
ISO 15512 : 1999	Plastics — Determination of water content
IS 15659 (Part 2) : 2006	Petroleum and natural gas industries — External coatings for burried or submerged pipelines used in pipeline transportation of gas and liquid hydrocarbons: Part 2 Fusion bonded epoxy coating

3 TERMINOLOGY

For the purposes of this standard, the following terms and definitions shall apply.

3.1 Applicator — The company in charge of the coating application in accordance with the provisions of this standard or the requirements given in the tender specification and/or in the order.

3.2 Batch — Discrete and continuous manufacturing operation using raw material of the same source.

3.3 Batch Certificate — Certificate of analysis issued by the manufacturer.

3.4 Certificate of Compliance — One of the types of documents to be issued in accordance with the purchase requirements.

3.5 Design Temperature — Maximum and minimum temperature likely to be reached during transport, handling, installation and operation.

3.6 End User — Company operating the pipeline system.

3.7 Manufacturer — Company responsible for the manufacture of coating material(s).

3.8 Manufacturer's Specification — Document, which specifies the characteristics, test requirements and application recommendations for the coating materials.

3.9 Purchaser — Company responsible for providing the product order requirements.

3.10 Operating Temperature — Normal or anticipated standard temperature, to be experienced by the pipe or pipeline system which should not exceed the design temperature.

3.11 Pipeline — Those facilities through which fluids are conveyed, including pipe, pig traps, components and appurtenances, up to and including the isolating valves.

3.12 Pipeline System — Pipeline with compressor or pump stations, pressure control stations, flow control stations, metering, tankage, supervisory control and data acquisition system (SCADA), safety systems, corrosion protection systems, and any other equipment, facility or building used in the transportation of fluids.

3.13 Total Coating Thickness — Sum of all three layers as defined in **8.1**.

3.14 Test Report — Document that provides the quantitative test results for tests conducted in accordance with the requirements of this standard.

3.15 Holiday — Coating discontinuity that exhibits electrical conductivity when exposed to a specific voltage.

3.16 Cut-Back — Length of pipe left uncoated at each end for joining purposes (for example, welding).

4 SYMBOLS AND ABBREVIATED TERMS

DSC — differential scanning calorimetric
ΔT_g — variation of the glass transition temperature ($T_{g2} - T_{g1}$), in °C
FBE — fusion bonded epoxy
HDPE — high density polyethylene
LDPE — low density polyethylene
M — nominal pipe mass per metre (kg/m)
MDPE — medium density polyethylene
MFR — melt flow rate
PE — polyethylene
PP — polypropylene
R_z — roughness parameter
SAW — submerged arc welding
T_g — glass transition temperature, in °C
3LPE — three layer polyethylene coating
3LPP — three layer polypropylene coating

5 GENERAL REQUIREMENTS

5.1 Compliance to Standard

A quality system should be applied to assist compliance with the requirements of this standard.

The applicator shall be responsible for complying with all of the applicable requirements of this standard. It shall be permissible for the purchaser to make any investigations necessary in order to be assured of compliance by the applicator and to reject any material that does not comply.

NOTE — IS/ISO 9001 gives guidance on the selection and use of quality systems and IS 14001/ISO 14001 gives guidance on the selection and use of environmental management systems.

6 CLASSIFICATION OF THE COATING SYSTEMS

6.1 General

The selection of classification depends on the expected field duty. The more aggressive the soil is, the higher shall be the coating thickness for each coating class. The transport, handling, laying conditions and the expected environmental conditions shall be considered as well for the thickness selection.

6.2 Coating Classes

The coating shall be capable of withstanding the temperature range required as shown in Table 1. The coating class shall be indicated in the purchase order.

Table 1 Coating Classes and Operational Temperatures

Sl No. (1)	Coating Class (2)	A (3)	B (4)
i)	Top layer material	MDPE/HDPE	PP
ii)	Operation temperature, °C	–40 to + 80	–20 to + 110

6.3 Coating Thickness Classes

The choice of coating thickness class is dependent on the soil or ground, laying method on land and offshore, service conditions and pipe dimensions. The coating thickness class as shown in Table 2 shall be indicated in the purchase order.

Classes A1, B1	are for moderate duty
Classes A2, B2	are for heavy duty such as rocky or clay soil, for example, a good water humidity barrier is required (submerged pipeline), an anti-rock system is not adopted or when storage, transport, handling, laying and service conditions are very severe (high temperature, harsh conditions, etc).

NOTE — It is recommended to obtain the coating thickness class from the end user.

7 INFORMATION TO BE SUPPLIED BY THE PURCHASER

7.1 Principal Information

The purchaser shall provide the following information:

a) Pipe material/grade; bare pipe standard or specification designation (IS 1978);
b) Pipe length, outside diameter and wall thickness, nominal pipe mass per metre;
c) Seam weld dimensions, if applicable;
d) Quantity of coated pipes;
e) Coating class;
f) Coating thickness class;
g) Design temperature (mandatory for Class B only); and
h) Type of certificate of compliance.

7.2 Supplementary Information

If applicable, the purchaser shall specify the following information:

a) Pipe tracking and traceability of pipes to coating materials;
b) Permissible number of coating repairs;
c) Marking of pipes;
d) Internal coating;
e) External/Internal temporary coating of pipes;
f) Colour of coating;
g) Handling of non-conformities (coated and non-coated pipes);
h) Documentation and schedule for supply of documentation;
j) Application procedure specification for purchaser's approval;
k) Inspection and testing plan and/or daily log for inspection and testing;
m) Inspection of incoming pipes (for example, checking of pipe dimensions and defects not affecting pipe coating);
n) Pipe end protection;
p) Minimum thickness of epoxy layer and total coating thickness exceeding those in Table 2 and Table 9 respectively;
q) Surface roughness of PE/PP coating [for example, concrete weight coating (CWC) or special laying methods];
r) Special requirements to supply of coating materials (for example, supplier specific products and certification);
s) Cut-back configuration other than specified in this standard; and
t) Methods, frequency and acceptance criteria for inspection and testing differing to this part of standard.

8 COMPOSITION OF THE COATING SYSTEM

8.1 General Requirements

The coating system shall consist of three layers:

a) 1 st layer : Epoxy
b) 2 nd layer : Adhesive
c) 3 rd layer : PE/PP top layer

The applicator shall identify the materials and shall confirm that the certificates comply and relate to the specified materials.

Table 2 Minimum Total Coating Thicknesses

(*Clauses* 6.3, 7.2 *and* 11.2.4)

Sl No.	Specified Outside Diameter mm	Coating Thickness mm			
		Class A1	Class A2	Class B1	Class B2
(1)	(2)	(3)	(4)	(5)	(6)
i)	Up to 273.1	2.1	2.7	1.5	1.9
ii)	273.1 to 508	2.5	3.1	1.8	2.3
iii)	508 to 813	2.8	3.5	2.2	2.5
iv)	813 and above	3.3	4.2	2.5	3.0

8.2 Pre-qualification of the Coating Materials

The manufacturer shall pre-qualify each type of coating material in compliance with the requirements of this standard. The pre-qualification shall be repeated in case of changes in the material composition, changes in the production process which influence the material processing behaviour and change in production facility.

The manufacturer shall carry out the material qualification in accordance with the basic requirements of **8** along with Table 3, Table 4 and Table 5. The test report shall contain the results of the qualification tests and the data required in Table 6, Table 7 and Table 9.

8.3 Epoxy Material

The applicator shall use epoxy material that is in compliance with Table 3.

8.4 Adhesive Material

The applicator shall use adhesive material that is in compliance with Table 4.

8.5 PE/PP Top Layer Material

The applicator shall use PE/PP material that is in compliance with Table 5.

The polyolefin shall be compounded and stabilized by additives against UV and thermal ageing. If the compounding is done during the application process the applicator shall qualify the process and carry out all the relevant tests *versus* the requirements of this part of the standard and shall certify that the compound is in compliance with this standard.

8.6 Material Data Sheet and Batch Certificate

The batch certificate shall be in accordance with Table 6.

8.6.1 Technical information concerning the epoxy materials provided by the manufacturer(s) shall be in accordance with the following:

a) Trade name;
b) Generic type;
c) Mix ratio (if any);

Table 3 Minimum Requirements for the Epoxy Material (FBE Powder)

(*Clauses* 8.2, 8.3 *and* 8.6.1)

Sl No.	Properties	Reference Standard/Annex	Requirements Class A	Requirements Class B
(1)	(2)	(3)	(4)	(5)
i)	Moisture content (by mass)	IS 15659 (Part 2)	≤ 0.6	
ii)	Minimum glass transition temperature (T_{g2}) (DSC analysis) (°C)	Annex D	≥ 95	+5 above operating temperature
iii)	Gel time	Annex L	Within 20 percent per manufacturer's specification	
iv)	Density in g/cm^3	IS 101 (Part 1/Sec7)	As per manufacturer's specification ± 0.05	
v)	Particle size	—	10-100 µm	

Table 4 Minimum Requirements for the Adhesive Material (Co-polymeric or Grafted Adhesive in Pellet or Powder Form)

(*Clauses* 8.2, 8.4 *and* 8.6.2)

Sl No.	Properties	Unit	Method of Test, Ref to	Requirements Class A	Requirements Class B
(1)	(2)	(3)	(4)	(5)	(6)
i)	Elongation at break at 23 ± 2°C	Percent	IS 13360 (Part 5/Sec 2)/ISO 527-2	≥ 600	≥ 400
ii)	Tensile yield strength at 23 ± 2°C	MPa	IS 13360 (Part 5/Sec 2)/ISO 527-2	≥ 8	≥ 12
iii)	Notched impact strength at minimum class temperature	kJ/m^2	IS 13360 (Part 5/Sec 5)/ISO 179	—	≥ 3
iv)	Vicat softening temperature A/50 (9.8 N)	°C	IS 13360 (Part 6/Sec 1)/ISO 306	100 min	≥ 110
v)	Water content	Percent	ISO 15512	≤ 0.1	≤ 0.1

NOTE — Two millimetre thick compression moulded sheet, test specimen type according to IS 13360 (Part 5/Sec 1)/ISO 527-2, strained at 50 mm/min (for PP based adhesives the specimen can be prepared also by injection moulding according to IS 10951).

Table 5 Minimum Requirements for PE/PP Top Layer

(*Clauses* 8.2, 8.5 *and* 8.6.2)

Sl No.	Properties	Unit	Test Method, Ref to	Requirements	
				Class A	Class B
(1)	(2)	(3)	(4)	(5)	(6)
i)	Density of the base resin[1]	g/cm^3	IS 13360 (Part 3/Sec 1)/ISO 1183	≥ 0.930	≥ 0.890
ii)	Elongation at break at 23 ± 2°C	Percent	IS 13360 (Part 5/Sec 2)/ISO 527-2	≥ 600	≥ 400
iii)	Tensile yield strength at 23 ± 2°C[2]	MPa	IS 13360 (Part 5/Sec 2)/ISO 527-2	≥ 15	≥ 20
iv)	Notched impact strength at minimum class temperature	kJ/m^2	IS 13360 (Part 5/Sec 5)/ISO 179	—	≥ 3
v)	Vicat softening temperature A/50 (9.8 N)	°C	IS 13360 (Part 6/Sec 1)/ISO 306	≥ 110	≥ 130
vi)	Water content	Percent	ISO 15512	≤ 0.05	≤ 0.05
vii)	Hardness Shore D	—	IS 13360 (Part 5/Sec 11)	≥ 55	≥ 60
viii)	ESCR (50°C, F50, Cond. A or B, 100 percent Igepal CO 630)[3]	h	ASTM D-1693	≥ 300 Cond. B	n.a.
ix)	Oxidation induction time (Intercept in the tangens method)	min	ISO 11357	≥ 30 at 210°C	≥ 30 at 210°C
x)	UV resistance and thermal ageing	—	Annex G	ΔMFR ≤ 35 percent	ΔMFR ≤ 35 percent
xi)	Service temperature	—	—	–40 to +80°C	–20 to+110°C

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[1] Preparation of the test specimen compression moulded sheet annealed for 30 min at 100°C.

[2] Two millimetre thick compression moulded sheet, specimen IS 13360 (Part 5/Sec 2)/ISO 527-2, strained at 50 mm/min (for PP the sample can be prepared also by injection moulding according to IS 10951).

[3] In order to validate the ESCR values, data shall be provided by manufacturers as to know the limit in density (MFR) to pass that requirement. This limit shall not be wider than what is commonly known as fit for purpose PE grades for pipe coating.

Table 6 Batch Certificate

(*Clauses* 8.2 *and* 8.6)

Content	Coating Material		
	FBE Primer	Adhesive	PE/PP Top
(1)	(2)	(3)	(4)
Identification of the manufacturer	x	x	x
Product identification	x	x	x
Batch identification	x	x	x
Date and place of manufacturing	x	x	x
Density	x	x	x
Melt flow rate		x	x
Gel time	x		
Particle size	x		
Moisture content	x	x	x
Carbon black content			Only PE
Pigment dispersion			x
Thermal characteristic	x		

d) batch certificate;
e) maximum thickness (mm) (*see* Annex A);
f) storage temperature, *Min* and *Max* (°C);
g) shelf life at storage temperature (months); and
h) technical properties (as per Table 3).

8.6.2 Technical information concerning the adhesive and PE/PP materials provided by the manufacturer(s) shall be in accordance with the following:

a) Trade name;
b) Description of the adhesive;
c) Colour;
d) Storage conditions;
e) Shelf life (months);
f) Technical properties (as per Table 4 and Table 5);
g) Batch No; and
h) Application temperature.

9 COATING SYSTEM QUALIFICATION

Each coating system shall be qualified by the applicator for each coating line. Applicator shall use coating materials already pre-qualified in accordance with the requirements of **8**. The minimum requirements and testing frequency of the applied coating are given in Table 9. The qualification shall be repeated in case of essential modifications of the coating line, coating materials and coating procedures.

10 PROPERTIES OF THE APPLIED COATING SYSTEM

The coating applied shall be in accordance with the requirements of Table 7.

11 APPLICATION OF THE COATING SYSTEM

11.1 Surface Preparation

Before blast cleaning the pipe surface shall be dry and free of any inorganic or organic contaminations (for example, soluble salts, oil, paint, grease, varnishes etc) detrimental to the surface preparation and coating. The soluble salts content shall not exceed 20 mg/m^2.

The surface shall be treated by suitable clean abrasives not contaminating the substrate. The degree of cleanliness must be Sa3 in accordance with ISO 8501-1.

The blasted surface shall have a angular roughness Rz of 50 to 100 or within the range proposed by the product manufacturer, measured with the stylus method according to ISO 8503-4, with a cut-off of 2.5 mm. Measuring method other than the stylus method may be used by agreement between the purchaser and the applicator.

After cleaning the surface of all pipes shall be free of steel defects, irregularities (for example, laminations, slivers, scratches, etc).

Grinding of defects shall not reduce the wall thickness below the specified minimum value. The maximum grinded area without re-blasting shall be limited to 10 cm^2/1 m pipe length.

Table 7 Properties of the Applied Coating

(*Clauses* 8.2, 10, 11.2.2 and *Annex* C)

Sl No.	Properties	Test Method	Class A	Class B
(1)	(2)	(3)	(4)	(5)
i)	Appearance and continuity	Annex B	Uniform colour, free of defects and discontinuities, delaminations, separations and pinholes	
ii)	Pipe end preparation		11.3	
iii)	Minimum impact strength at 23°C (J/mm)	Annex E	> 7	> 10
iv)	Indentation (mm) at 23°C at maximum class temperature	Annex F	≤ 0.2 ≤ 0.4	≤ 0.1 ≤ 0.4
v)	Elongation at break of the PE/PP (23 ± 3°C)	IS 13360 (Part 5/Sec 2)/ ISO 527-2	≥ 300 percent	≥ 300 percent
vi)	Peel strength[1] (N/mm)	Annex C	≥ 15 at 23°C ± 1°C ≥ 3 at 80°C[3]	≥ 25 at 23°C ± 1°C ≥ 4 at 90°C or at maximum operating temperature if above 90°C[3]
vii)	Degree of cure of the epoxy (1st layer)	Annex D	ΔT_g +3°/–2°C[2] Scratch test for liquid epoxy	
viii)	Product stability during extrusion of the PE/PP top layer process	IS 13360 (Part 4/Sec 1)/ ISO 1133	≤ 20 percent ΔMFR for class A; ≤ 35 percent for class B (virgin compounded granulate before extrusion/extruded foil after extrusion of the same batch)	
ix)	Average radius of cathodic disbondment at: 23°C/28 days; –1.5 V or 65°C/24h; –3.5 V Maximum operation temperature/28days/–1.5 V	Annex H	 ≤ 7 mm ≤ 7 mm ≤ 15 mm	
x)	Flexibility at 0°C	Annex J	2 percent strain	
xi)	Hot water soak test	Annex K	No loss of adhesion	

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[1] No single recorded peeling value shall be lower than 30 percent of the requirements.

[2] No disbonding between steel and epoxy.

[3] For FBE with a glass transition temperature above 120°C the material manufacturer shall recommend the test method.

The pipe surface shall be cleaned of residual dust and contaminants by approved suitable cleaning methods. The dust contamination shall not exceed Grade 2 in accordance with ISO 8502-3.

Chemical treatment/chromate conversion coating of the pipe surface may be carried out, if not excluded in the purchase order.

The pipe temperature prior to the heating to the application temperature shall be at least 3°C above the dew point.

11.2 Coating Application

11.2.1 *General*

The coating shall be applied in accordance with the procedures approved during the qualification of the applicator.

During the application of the coating components, the preheating temperature of the pipe shall be checked and recorded.

Temperature shall be monitored and recorded continuously using optical pyrometers or contact thermometers. The use of the tempil sticks shall be agreed and verified during qualification of the coating system.

Once the coating temperature is established, the temperature of at least every fifth pipe shall be taken and recorded.

11.2.2 *Epoxy Application*

The elapsed time between surface preparation by abrasive blasting, cleaning and coating application depends on the environmental conditions in the coating plant and shall be adequate to avoid oxidation or contamination of the pipe surface.

The pipe temperature prior to the epoxy application and during application shall be in accordance with the coating parameters of the qualifications. The thickness of the epoxy layer (*see* Table 9) after curing shall be in accordance with the coating parameters of the qualifications and capable to satisfy the requirements of Table 7.

The rate of curing shall be checked in compliance with Table 7.

11.2.3 *Adhesive Application*

The elapsed time between epoxy application and adhesive application shall be in accordance with the coating parameters of the qualifications and the requirements of Table 9.

11.2.4 *PE/PP Application*

The application method shall be lateral or cross-head extrusion.

The extrusion conditions and the mass temperature of the extruded material shall be in accordance with the coating parameters of the qualifications.

The applied coating shall be cooled down to a temperature which prohibits damages caused by transportation and handling of the coated pipe during finishing and final inspection. The maximum coating temperature after cooling shall be stated by the applicator.

The total coating thickness shall be in accordance with Table 2.

11.3 Pipe End Preparation

The coating at the pipe ends (cut back) shall be removed over a length depending on the nominal pipe diameter in accordance with Table 8 (unless otherwise agreed).

Table 8 Cut Back Length

Sl No. (1)	Pipe Diameter mm (2)	Cut Back Length mm (3)
i)	≤ 500	100 ± 10
ii)	≥ 500	150 ± 20

The coating shall be bevelled by suitable methods to an angle between 20° and 30° measured in direction to the pipe axis. The cut back length shall be measured beginning of the root face of the pipe to the beginning of the coating bevel. If the top layer is applied by cross-head extrusion, precautions shall be taken to avoid disbondment on the cut back. In front of the coating bevel the epoxy shall be visible.

12 COATING REPAIRS

Localized defects in the finished coating caused by the application process, transportation, handling and storage in the coating plant or in the storage area as well as those which have been subjected to destructive testing shall be repaired.

The maximum defected area shall be 10 cm^2 and the total amount of defects to be repaired shall not exceed 2 defects per pipe of the coated surface.

The repair procedure and the repair material shall be compatible with the applied coating. The applicator shall install and maintain repair and testing procedures. These repair procedures shall be part of the coating system qualification.

13 INSPECTION AND TESTING

The inspection and testing shall be carried out in accordance with the requirements of Table 9.

Table 9 Requirements for Coating System Qualification, Inspection and Testing During Production

(*Clauses* 7.2, 8.2, 9, 11.2.3 *and* 13)

Sl No.	Properties	Test Method	Requirements	Frequency for Qualification Test	Frequency During Production
(1)	(2)	(3)	(4)	(5)	(6)
i)	Surface condition before blasting	Visual inspection	Free of contaminations salt	Each pipe	Each pipe
	Surface condition after blasting	Conductive measurement	Content maximum 20 mg/m^2 ISO 8502-9	Each pipe	5 pipes at start of production and 1 pipe/shift
ii)	Environmental conditions (dew point)	Calculation	Relative humidity maximum 85 percent	Once	Every 4 h
iii)	Pipe temperature before blasting	Thermocouple	Minimum 3°C above the dew point	Once	Every 4 h
iv)	Size, shape and properties of abrasive	Visual + certification ISO 11124 resp. ISO 11126	Conformity to certificate	Once	1/day
v)	Water soluble contamination of abrasives	ISO 11127-6	Salt content maximum 20 mg/m^2 ISO 8502-9	Once	1/shift
vi)	Surface roughness of blasted surface (R_z/R_{y5})	ISO 8503-4	50 µm to 100 µm	Each pipe	Each pipe
vii)	Visual inspection of blasted surface	ISO 8501-1	Grade Sa3	Each pipe	Each pipe
viii)	Presence of dust after dust removal	ISO 8502-3	Maximum class 2	5 pipes	Every 1 h
ix)	Elapsed time between blasting and coating	Monitoring	No rust, pipe temperature at least 3°C above the dew point	Continuously	Continuously
x)	Temperature of extruded adhesive and polyolefin	Thermometer	Compliance to coating procedure	Once	Every 1/h
xi)	Preheating temperature before coating	Thermometer	Compliance to manufacturing procedure specification	Continuously	Continuously
xii)	Minimum epoxy thickness	IS 101 (Part 3/Sec 2)	Liquid epoxy: 25 µm FBE: 150 µm	At start up	Each start up
xiii)	Minimum adhesive thickness	—	200 µm on pipe body for SAW welded pipes	At start up	Each start up
xiv)	Degree of cure	Annex D ISO 11357-2	$\Delta T_g \leq +3°/-2°C$ for FBE scratch test for liquid epoxy	4 Pipes from partly epoxy and adhesive coated and 4 pipes from fully (all three layer) coated pipe and at start up	One/shift and at each start up
xv)	Appearance and continuity	Visual Annex B	Table 6	Continuously	Continuously
xvi)	Total thickness of coating	Annex A	Table 2	Each pipe	Every 10 pipes
xvii)	Impact resistance	Annex E	Table 7	3 pipe	Once per 2 weeks
xviii)	Peel strength	Annex C	Table 7	2 pipe	Every 4 h
xix)	Indentation	Annex F	Table 7	Once	Each PE/PP batch
xx)	Elongation at break	IS 13360 (Part 5/ Sec 2)/ISO 527-2	Table 7	Once	Each PE/PP batch
xxi)	Cathodic disbondment	Annex H	Table 7	Once for each batch	Once in a week
xxii)	Flexibility	Annex J	Table 7	Once	—
xxiii)	Hot water soak test	Annex K	Table 7	Once	—
xxiv)	In process degradation of PE/PP	IS 13360 (Part 4/ Sec 1)/ISO 1133	ΔMFR maximum 35 percent for PP; 20 percent for PE between raw and extruded material	Once	At start up
xxv)	Pipe end preparation	Measuring	11.3	Each pipe	Each pipe
xxvi)	Coating repairs	Visual/Holiday detection	No holidays	Once for demonstration	Each defect

Pipes which failed the test shall be re-tested. In case the re-test failed two pipes in the coating sequence prior of the pipe which failed and two pipes in the sequence after that pipe shall be re-tested.

If the results of all re-tested pipes are satisfactory, the coating shall be considered acceptable excluding the pipe which failed. If the tests fail, the total production back to the last passed test shall be blocked and further testing shall be initiated by the applicator.

14 HANDLING, TRANSPORTATION AND STORAGE IN THE COATING AREA

Coated pipe shall be handled in a manner that avoids damage to the pipe and coating. Where specified in the purchase order, the applicator shall submit details of the handling procedures; such procedures shall include loading requirements where the applicator is responsible for loading.

If specified in the purchase order, the applicator shall submit details of the facilities and the methods to be used for yard storage.

15 MARKING

The coating shall be legibly and indelibly marked with the following:

a) Applicator's name or code;
b) Reference to this standard;
c) Coating class; and
d) Coating thickness.

Example: Applicator — IS 15659 (Part 1) class B2 –2.5 mm, operating temperature XXX °C

15.1 BIS Certification Marking

The product may also be marked with the Standard Mark.

15.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

ANNEX A

[*Clause* 8.6 *and Table* 9, *Sl No.* (xvi)]

INSPECTION OF THICKNESS

A-1 GENERAL

The test consists of measuring, by means of a non-destructive process, the thickness of the applied coating.

A-2 INSTRUMENT

A magnetic, electromagnetic or ultrasonic measuring instrument with ± 10 percent accuracy shall be used. The instrument shall be for the range of coating thickness to be measured and shall be calibrated.

A-3 PROCEDURE

The instrument shall be verified on a steel pipe surface using a calibrated shim/foil. The surface profile and cleanliness of the steel pipe surface shall be representative of the production pipe.

For measuring thickness of more than 1 mm, the surface profile of the pipe is not relevant.

On each pipe to be tested, the total of 12 measurements shall be carried out in accordance with IS 101 (Part 3/ Sec 2). Every measurement consists of the average of 5 single readings in the defined area. All readings must meet the minimum requirements.

In case of welded pipes 4 readings shall be made at the apex of weld seam, uniformly distributed over the length of coated pipe.

The measurements areas shall be distributed along 4 equally spaced longitudinal lines at the pipe length with three equally circumferential lines on the middle of the pipe and at a distance of at least 300 mm from the end of the coating.

A-4 RESULTS

The arithmetic average of all measurements shall be calculated and recorded.

ANNEX B

(*Tables* 7 *and* 9)

HOLIDAY DETECTION TEST

B-1 GENERAL

For the test detecting any pinhole or other defects in the coating a scanning electrode energized by a high-arc-voltage shall be used.

Defects shall be detected by a spark occurring between the steel of the pipe and the electrode at the defect, accompanied by a sound and light signal provided by the holiday detector.

B-2 EQUIPMENT

The equipment shall consist of,

a) adjustable high-voltage holiday detector, equipped with a sound and light signal;

b) scanning electrode in the form of a metal brush, or conductive rubber conforming to the shape of the pipes; and

c) conductors which are used to connect the pipe to the holiday detector.

B-3 PROCEDURE

The instrument shall be connected to the pipe, completing the circuit, switched on, and the scanning electrode passed over the surface of the coating to be inspected with a continuous movement. The rate of the relative movement of the electrode shall not be limited but it shall be demonstrated that a defect of 1 mm diameter will be detected. Travel speed shall not exceed 300 mm/s.

At the time of the test, the voltage shall be set at 10 kV/mm of the minimum thickness of coating and shall not exceed 25 kV.

B-4 RESULTS

The number of holidays detected shall be recorded.

ANNEX C

(*Tables* 7 *and* 9)

PEEL STRENGTH TEST

C-1 GENERAL

The method consists of measuring the force required for peeling the coating from the metal substrate of the pipe at a constant rate of pull (*see* Fig. 1).

C-2 EQUIPMENT

The equipment shall consist of:

a) tensile testing machine with which it is possible to record the peel force and which operates at a rate of pull of 10 mm/min, as in Fig. 1 or Fig. 2;

b) cutting tool (for example, knife).

C-3 PROCEDURE

The test shall be performed at temperatures in accordance with Table 7.

The temperature shall be measured by means of an adapted probe, on the external surface of the pipe at the root of the peeled strip.

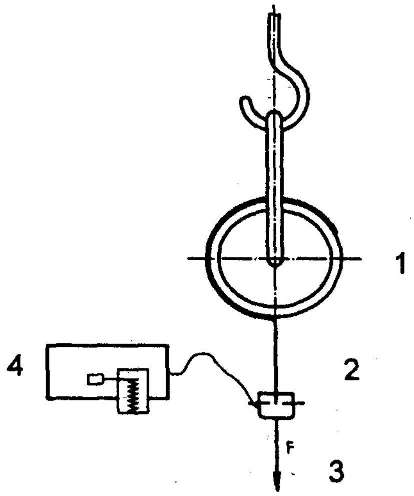
A sample of minimum length of 160 mm shall be cut in the circumferential direction from the coated pipe. From this sample, a strip of coating 20 mm to 50 mm wide perpendicular to the axis of the pipe shall be peeled. First 20 mm and last 20 mm shall not be counted for assessment of bond strength.

The pipe shall be arranged on its support and shall be secured to one of the gripping jaws of the testing machine. The system shall be such as to allow the reciprocal movement between pipe and testing machine at the prescribed pulling speed.

The peel force shall be graphically recorded over the agreed length using a constant peeling speed set to 10 mm/min.

C-4 RESULTS

The peel strength shall be calculated from the recorded data divided in five sections of 20 mm each. The average peel strength shall be calculated for each of these sections.

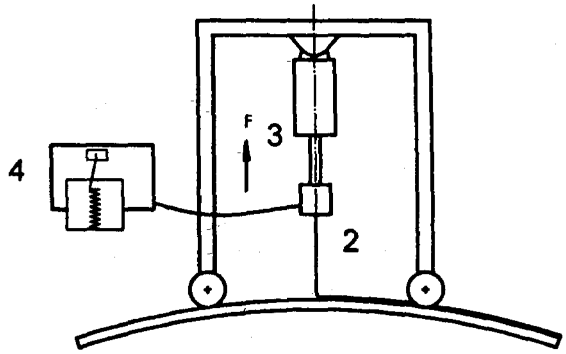


Key

1	Pipe ring	3	Peel force
2	Coating strip	4	Registration unit

FIG. 1 TESTING EQUIPMENT (SMALL DIAMETERS)

The average peel strength shall be the arithmetic mean taken over 100 mm length. Where this value is not automatically determined, the arithmetic mean may be estimated from the 20 mm bands across the 100 mm length.



Key

1	Coated pipe surface	3	Peel force
2	Coating strip	4	Registration unit

FIG. 2 TESTING EQUIPMENT (LARGE DIAMETERS)

The system shall disbond/separate cohesively either in adhesive layer or in polyethylene layer. Disbondment/separation at epoxy to steel interface or epoxy/adhesive interface or adhesive/polyethylene interface shall not be permitted. The failure mode shall be recorded for each test.

The minimum average shall be the lower value from the following two determinations:

a) Lowest arithmetic mean from each of the 20 mm sections shall meet the requirements of Table 7; and
b) No single point in each of the 20 mm sections shall be lower than 30 percent of the specified value of Table 7.

ANNEX D

(*Tables 7 and 9*)

THERMAL ANALYSIS OF EPOXY POWDER AND CURED COATING FILM (FBE)

D-1 GENERAL

By thermal analysis the uncured epoxy powder and the cured coating film are characterized.

The method to be used is Thermal-Scanning-Calorimetry (DSC). The general procedure and definitions are given in ISO 11357-1. Reference is made to this standard. General handling and calibration shall be performed as in ISO 11357-1 unless stated differently in this method.

D-2 EQUIPMENT

The equipment shall consist of the following:

a) Differential-scanning-calorimeter (DSC) with cooling accessory;
b) Balance accurate to 0.1 mg;
c) Sample encapsulating press;
d) Aluminium pans with covers; and
e) Dry gas supply of N_2, analytical grade.

D-3 PROCEDURES AND MEASUREMENT FOR EPOXY POWDER

D-3.1 Measurement

The measurement shall consist of the following steps:

a) Heat the sample from 25 ± 5°C to 70 ± 5°C at a rate of 20°C/min, then immediately cool the sample to 25 ± 5°C.
b) Heat the sample from 25 ± 5°C to 275 ± 5°C at a rate of 20°C/min, then immediately cool the sample to 25 ± 5°C.

c) Heat the sample from 25 ± 5°C to $T_g + 40°C$ (typically 150°C) at a rate of 20°C/min, then immediately cool the sample to 25 ± 5°C.

For certain epoxy powders different heating cycles might be necessary according to instructions of the epoxy powder manufacturer.

D-3.2 Evaluation of Results

D-3.2.1 *Glass Transition Temperature,* T_g

The T_g is calculated at the point of inflection intersection (Fig. 3).

By evaluating run (b) the T_g of the uncured powder is obtained = T_{g1}. By evaluating run (c) the T_g of the cured material is obtained = T_{g2}.

D-3.2.2 *Heat of Reaction of Epoxy Powder*

The exothermic heat of reaction (ΔH) is obtained by integrating the exothermic peak of the DSC-scan.

D-4 PROCEDURES AND MEASUREMENT FOR COATING SAMPLE

D-4.1 General

A representative sample of the cured film is taken.

Weigh in 10 ± 3 mg to an accuracy of 0.1 mg. The pan is sealed with the cover. Determine final weight after sealing.

Place the sample and the reference sample in the DSC cell and purge with dry, N_2 gas.

D-4.2 Measurement

The measurement shall consist of the following steps:

a) Heat the sample from 25 ± 5°C to 110 ± 5°C at a rate of 20°C/min and hold for 1.5 min, then cool the sample to 25 ± 5°C.

b) Heat the sample from 25 ± 5°C to 275 ± 5°C at a rate of 20°C/min, then cool the sample to 25 ± 5°C.

c) Heat the sample from 25 ± 5°C to $T_g + 40°C$ (typically150°C) at a rate of 20°C/min, then cool the sample to 25 ± 5°C.

For certain epoxy powders different heating cycles might be necessary according to instructions of the epoxy powder manufacturer.

NOTE — Samples taken from pipes, which have been stored or buried, shall be dried before testing.

D-4.3 Evaluation of Results

D-4.3.1 *Glass Transition Temperature,* T_g

The T_g is calculated in the same way as for the epoxy powder for run 3 (b) and 3 (c) (Fig. 4).

where

T_g = T_g of run 3(b) (°C)

T_{g4} = T_g of run 3(c) (°C)

For coatings, determine the change in T_g value using the following formula:

$$\Delta T_g = T_{g4} - T_{g3}$$

where

ΔT_g = change in T_g value (°C)

D-4.3.2 *Residual Heat of Reaction of Cured Coating*

The exothermic heat of reaction is obtained by

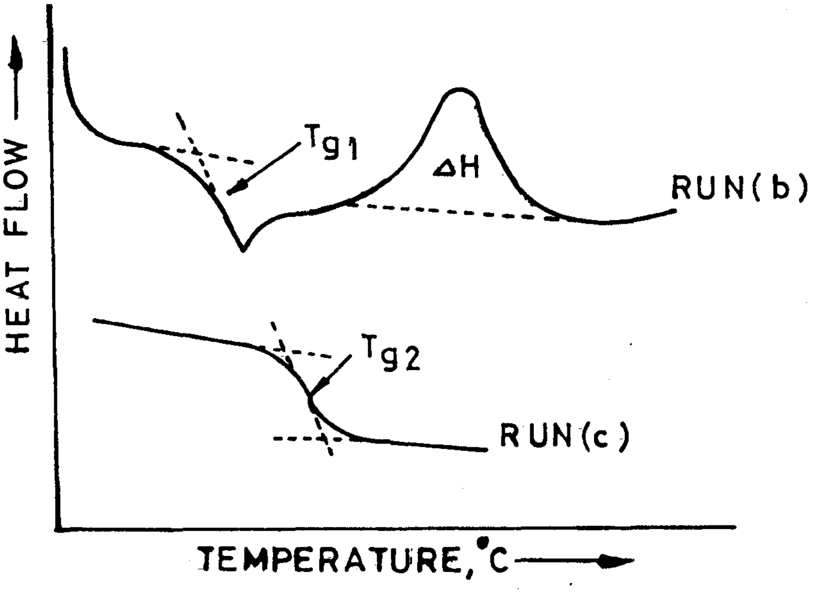


FIG. 3 EXAMPLES OF THERMAL SCANS ON EPOXY POWDER

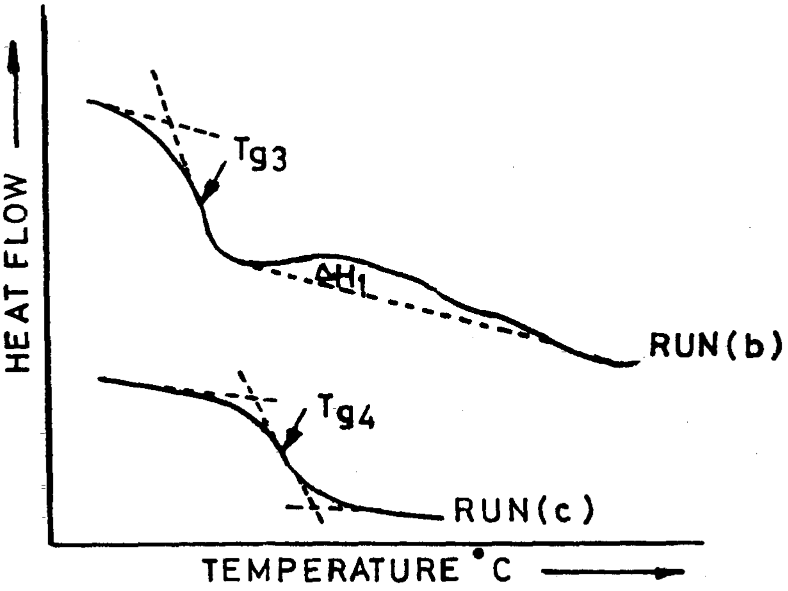


FIG. 4 EXAMPLES OF THERMAL SCANS ON COATING

integrating the exothermic peak of the DSC-scan 4 b; $= \Delta H_1$.

In a fully cured coating film there should be no residual heat of reaction be visible.

The degree of conversion C can be calculated by:

$$C = \frac{\Delta H - \Delta H_1}{\Delta H \times 100}$$

where

C = degree of conversion (percent),

ΔH = exothermic heat of reaction of the powder (run 3 b), and

ΔH_1 = exothermic heat of reaction of the coating film (run 4 b).

D-5 RESULTS

The following information shall be recorded:

a) Type of material and batch number;

b) Date of testing;

c) Type of DSC equipment;

d) For the epoxy powder T_{g1}, T_{g2}, ΔH;

e) For the cured coating film T_{g3}, T_{g4}, ΔT_g, ΔH_1 and C.

Testing of production coating requires pipe number or identification.

ANNEX E

(*Tables* 7 *and* 9)

IMPACT TEST

E-1 GENERAL

The test consists of verifying the strength of the coating by the impact of a punch of defined shape falling directly on to the coating from a fixed height and at a fixed temperature. The test shall be carried out on pipes or cut samples. This test shall not be carried out on pipes with a diameter of less than 50 mm.

E-2 DROP WEIGHT EQUIPMENT

The equipment shall consist of a drop weight testing machine comprising:

a) Straight guide made of steel, aluminium or plastic, rigid and non-deformable, of inside diameter between 40 mm to 60 mm, of length at least 1.30 m and incorporating a smooth and even inside surface. The guide shall be provided with:

    1) support and levelling devices (for example, two spirit levels for the horizontal plane and a plumb line for the vertical plane);
    2) graduated rod which makes it possible to determine the drop height to an accuracy of 5 mm.

b) Hard steel punch, with a hemispherical head, free from notches, porosity or other surface irregularities and with a diameter of 25 mm. A small metal rod of diameter 6 mm shall be fixed perpendicular to the flat face of the head and in its centre, where this rod shall be long enough to hold the additional weights required for the tests. The punch shall be equipped with a suitable system for raising to the required height; the mass of this assembly shall be 1 ± 0.005 kg; and

c) Sufficient number of additional weights, formed by metal discs (preferably made of stainless steel) with an outside diameter fitting the internal diameter of the straight guide 24 mm and incorporating a central hole of a suitable diameter; the mass of each disc shall be with an accuracy of ± 5 g.

NOTE — Other guides may be used by agreement.

E-3 PROCEDURE

The test shall be carried out at a temperature of 23 ± 3°C. If provisions have been made to perform this test outside this temperature range, the method described shall be adapted, after agreement, if necessary, between the coater and the purchaser.

The coated pipe shall be placed on a rigid, and stable, horizontal support, and if necessary, the pipe interior shall be supported to reduce its elastic response.

Before carrying out an impact test, the holiday detection test shall be performed (*see* Annex B) to identify the defective points and to avoid making the impact at these locations.

For each point of impact, the drop weight testing machine shall be installed perpendicular to the coating surface so that the loaded punch can fall freely without friction or resistance.

E-3.1 Basic Method

Ten impacts shall be carried out allowing the weight corresponding to the energy laid down in **10** and Table 7 to fall from a height of 1 m. The points of impact shall be selected and shall avoid any protruding welds. Further more, the distance from the points of impact to the end of the pipe shall be at least 1.5D (D is the outside diameter of the pipe) and at least 50 mm apart between the axes of the impacts. The holiday detection test shall then be undertaken at each location (*see* Annex B).

E-3.2 Alternative Method

Depending on the result obtained (number of perforations obtained from the ten impacts made), a further series of ten is carried out with increasing or decreasing weights, so as to be able to plot a curve of the number of perforations with respect to the impact energy. The point of inflection is determined, with a required accuracy of 1 Joule per mm of coating thickness. The point of inflection is that part of the curve where there is a rapid increase in the number of perforations (in practice five or six well distributed tests are adequate). The hard steel punch shall be checked every 30 impacts. If damaged, it shall be replaced.

E-4 RESULTS

If no holiday are recorded on any of the ten impacts using the basic methods, the impact results are accepted. The alternative method gives the maximum impact energy, in Joule per mm, to which the coating might be expected to withstand and is defined as the point of inflection of the graph.

ANNEX F

(*Tables* 7 *and* 9)

INDENTATION TEST

F-1 GENERAL

The test consists of measuring the indentation of a punch into the coating under fixed conditions of temperature and load.

F-2 INDENTATION TEST EQUIPMENT

The equipment shall consist of:

a) inside ventilated oven or a circulated bath thermostatically controlled to ± 2°C;

b) penetrometer comprising:

   1) cylindrical punch with a diameter of 1.8 mm (cross-sectional area 2.5 mm^2) on the top of which is mounted a weight. The assembly, punch plus weight, shall produce a force of 25 N; and

   2) dial gauge or any other measurement system accurate to ± 10^{-2} mm.

F-3 PROCEDURE

The test shall be performed three times on one PE/PP coating sample.

The test sample, held within the penetrometer assembly, shall be placed in the thermostatically controlled chamber and set to the test temperature. The test sample shall be kept in the chamber for 1 h. The readings on the dial gauge shall be recorded.

The shock resistant punch with the mass of 2.5 kg in total shall be loaded to the equipment. The test duration shall be 24 h. The readings of the dial gauge shall be recorded.

F-4 RESULTS

The indentation shall be the difference between the dial gauge reading before and after the 24 h test duration. The average of the 3 samples shall meet the requirements of Table 7.

ANNEX G

(*Table* 5)

UV-AGEING TEST AND THERMAL AGEING TEST

G-1 UV-AGEING

G-1.1 General

The test consists of subjecting PE/PP material test samples to the continuous irradiation of a xenon lamp under given temperature and humidity conditions.

The change in the PE/PP material shall be assessed by the variation in its elongation at break or in its melt flow rate.

G-1.2 Equipment

The equipment shall consist of an irradiation chamber equipped with a xenon lamp (*see* ISO 4892-2).

G-1.3 Sampling

The test shall be carried out on a sample of PE/PP in accordance with ISO 1872-2 for PE and IS 13360 (Part 5/Sec 2)/ISO 527-2 for PP.

G-1.4 Procedure

The size of the sample shall be large enough to make 10 test specimens of Type 1B, 5A or 5B [*see* Figs. 1 and 2 in IS 13360 (Part 5/Sec 2)/ISO 527-2].

The test specimen shall be exposed under the following conditions:

a) Artificial weathering (*see* Table 1 — Method A in ISO 4892-2);

b) Black standard temperature BST 65 ± 3°C;

c) Relative humidity 65 ± 5 percent;

d) Spray cycle: 18 ± 0.5 min spray/102 ± 0.5 min dry;

e) Total radiant energy 7 GJ/m^2 for class A, 5 GJ/m^2 for Class B; and

f) Continuous exposure.

The elongation at break of five irradiated test specimen shall be measured in accordance with IS 13360 (Part 5/Sec 2)/ISO 527-2.

Instead of the elongation at break method, three melt flow rate measurements can be carried out in accordance with IS 13360 (Part 4/Sec 1)/ISO 1133.

G-1.5 Results

G-1.5.1 *Elongation at Break*

The elongation (A_n) for each specimen shall be expressed as a percentage using following formula:

$$A_n = \frac{L_1 - L_0}{L_0} \times 100$$

where

L_0 = initial distance between the gauge marks on the central part of the test specimen, expressed in mm; and

L_1 = distance between the gauge marks at break, expressed in mm.

The arithmetic mean (A) shall be expressed as a percentage using following formula:

$$A = \frac{A_1 - A_0}{A_0} \times 100$$

where

A_0 = arithmetic average of five single values before exposure (*see* Annex H) (percent), and

A_1 = arithmetic average of five single values after exposure (percent).

G-1.5.2 *Melt Flow Rate*

The variation of the melt flow rate (ΔMFR) after exposure shall be expressed as a percentage using following formula:

$$\Delta MFR = \frac{MFR_1 - MFR_0}{MFR_0} \times 100$$

where

MFR_0 = initial melt flow rate measured before exposure, and

MFR_1 = melt flow rate measured after exposure.

G-2 THERMAL AGEING

G-2.1 General

The test consists of subjecting PE/PP material test samples to the effect of dry heat from a thermostatically controlled oven.

The change in the PE/PP material shall be assessed by the variation in its melt flow rate.

G-2.2 Equipment

The equipment shall consist of a thermostatically controlled oven with air circulation maintaining a test temperature within ± 2°C.

G-2.3 Sampling

The test shall be carried out on a sample of PE/PP material in accordance with ISO 1872-2 for PE and IS 13360 (Part 5/Sec 2)/ISO 527-2 for PP.

G-2.4 Procedure

The test shall be carried out at the temperature of 100 ± 2°C for classes A and B.

The test sample shall be placed in an oven set to this temperature and maintained for an exposure time of 2 400 h for class A and 4 800 h for class B.

Three melt flow rate measurements shall be undertaken on the test sample in accordance with IS 13360 (Part 4/Sec 1)/ISO 1133.

The arithmetic mean of the results obtained MFR_1 shall be calculated. The melt flow rate MFR_0 shall be calculated in accordance with IS 13360 (Part 4/Sec 1)/ISO 1133 on a test sample of identical shape which has not been placed in the oven.

G-2.5 Results

The variation of the melt flow rate (ΔMFR) after 2 400 h shall be expressed as a percentage using following formula:

$$\Delta MFR = \frac{MFR_1 - MFR_0}{MFR_0} \times 100$$

where

MFR_0 = initial melt flow rate measured before exposure, and

MFR_1 = melt flow rate measured after exposure.

ANNEX H

(*Tables* 7 *and* 9)

CATHODIC DISBONDMENT TEST

H-1 GENERAL

The test consists of assessing the resistance to disbondment of damage to the coatings when exposed to cathodic polarisation.

The test shall be performed on test specimen taken from the coated components previously subjected to holiday-detection (*see* Annex B), in which an artificial defect of a defined size has been drilled. The test may be performed on the coated component without cutting test specimens.

H-2 EQUIPMENT

H-2.1 Electrical Source

The source for the voltage and the current shall consist of a stabilized d.c. power unit. The current source shall be capable of supplying 20 mA to each test area simultaneously. A cathodic polarisation potential of –1 500 mV to a saturated calomel reference electrode (equivalent to UH = –1 260 mV where UH means the potential of the standard hydrogen electrode) shall be maintained (*see also* **H-2.3**).

H-2.2 Electrolytic Cell

For tests, typical test cell configurations are shown in Fig. 5 for large diameter components and in Fig. 6 for small diameter components.

The electrolytic cell shall comprise of:

a) rigid plastic pipe of an internal diameter of minimum 50 mm. The height shall be such that the total volume of the electrolyte is equal or greater than 150 ml with a minimum height of the electrolyte of 70 mm; and
b) rigid plastic cover in which holes shall be drilled to allow the passage of the electrodes and any other measuring instruments deemed necessary, and to allow the escape of hydrogen.

H-2.3 Electrodes

H-2.3.1 *Reference Electrode*

The reference electrode suitable to produce a potential (*see* **H-2.1**) shall be placed in an electrode holder situated in a glass pipe with a porous end diaphragm. The end of this assembly shall be placed approximately 10 mm from the surface of the coating and approximately 20 mm from the coating defect (Fig. 5). The reference electrode used shall be suitable for the test temperature required.

H-2.3.2 *Auxiliary Electrode (Anode)*

The auxiliary electrode shall consist of an inert material, that is platinum wire of 0.8 mm to 1.0 mm diameter. It shall be immersed in the electrolyte (approximately within 10 mm over the coating defect).

H-2.3.3 *Working Electrode (Cathode)*

The working electrode is represented by the artificial defect which shall be 6 mm in diameter, with a maximum depth of 0.5 mm in the steel substrate (*see* **H-3** and Fig. 7).

The ratio of the surface area of the anode and the cathode (surface of artificial defect) shall be greater than 1.

H-2.4 Electrolyte

The electrolyte shall consist of a solution of 3 percent NaCl concentration in distilled or deionized water. The solution shall be made from annular grade sodium chloride.

The *p*H at the 23 ± 2°C during the test shall be in the range of 6 to 9.

The height of the electrolyte in the cell shall be 75 mm ± 5 mm.

H-2.5 Heating Equipment

Suitable heating equipment shall be used to establish and to maintain the test temperature of the sample.

If not heated in an oven the temperature shall be checked on the steel surface by an appropriate means for example, a temperature sensor.

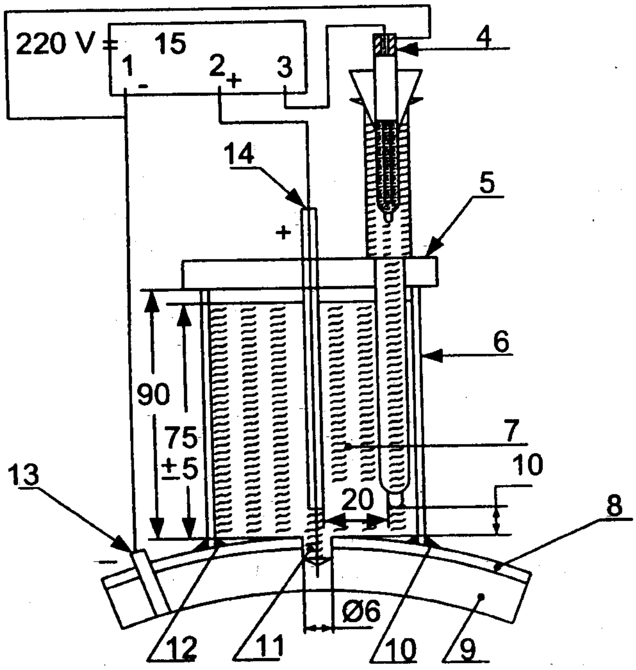
H-3 SAMPLING

The test samples shall be cold cut from a coated component and shall have a minimum size of 80 mm × 80 mm, unless the test is performed on the body of the coated component.

Test samples shall not be taken from the weld area.

For each sample the thickness of the area of the coating subject to the test shall be measured and recorded. The integrity of the coating on all test samples shall be checked by holiday detection (*see* Annex B).

A 6 mm diameter hole (*see* Fig. 7) shall be drilled



Key

1 Working electrode
2 Electrode (anode)
3 Electrode (reference)
4 Reference electrode
5 Plastic cover
6 Plastic pipe, minimum internal Ø 50 mm
7 Electrolyte ≥ 150 ml
8 Coating
9 Steel test piece
10 Sealing material
11 Artificial defect
12 Sealing material
13 Electrode (cathode)
14 Platinium electrode Ø 0.8 mm to 1.0 mm (anode)
15 Potentiostat

Fig. 5 Electrolytic Cell for Large Diameter Pipe

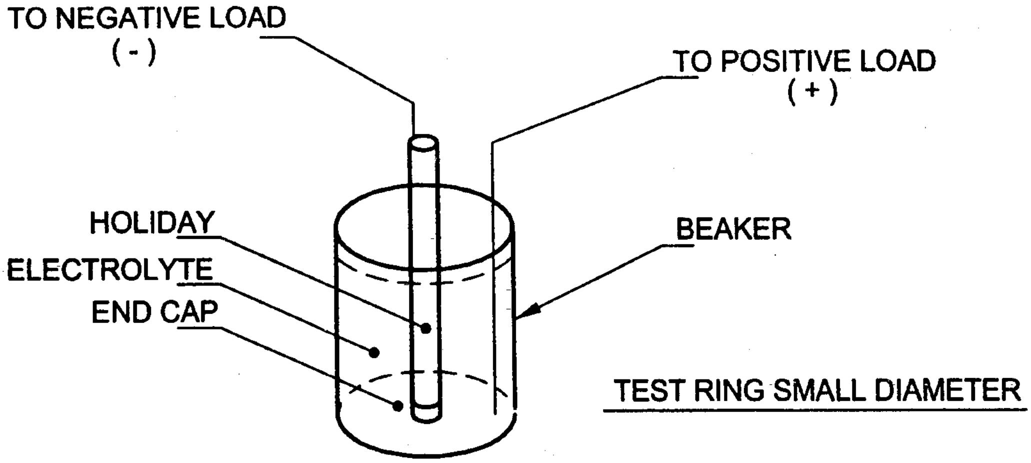


Fig. 6 Electrolytic Cell for Small Diameter Pipe

Key

1 Fluted and mill face mill Ø 6 mm
2 Conic end
3 Coating
4 Steel

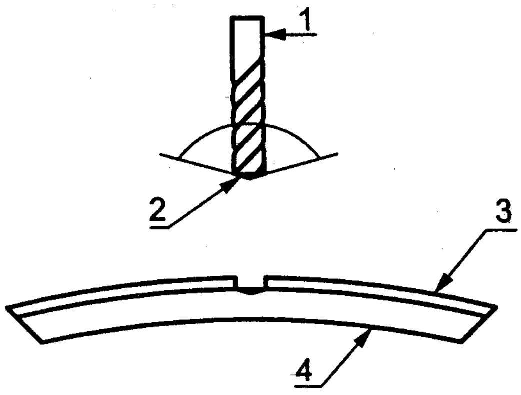


Fig. 7 Drilling of Artificial Defect

through the coating in the centre of the test sample using a standard drill bit. The depth of the drilled hole in the steel substrate shall not exceed 0.5 mm. At the initiation of the test the total surface area subject to the test shall be free from residual coating.

The test area shall be degreased using a suitable solvent and then rinsed with potable water and subsequently dried.

H-4 PROCEDURE

The plastic pipe forming the electrolytic cell shall be sealed using a suitable sealant, that is, a chemically inert adhesive. The artificial defect shall be in the centre of the cell.

The cell shall be filled with the NaCl electrolyte (*see* **H-2.4**).

A negative cathodic potential shall be applied between the reference and working electrodes (*see* Fig. 5 or Fig. 6), with an accuracy of ±10 mV. If a saturated calomel electrode is used the potential shall be –1 500 mV.

The test shall be performed for the test period required. The level of the electrolyte shall be readjusted with distilled or deionized water, if necessary.

H-5 INVESTIGATION PROCEDURE

After the test, the cell with the electrolyte shall be removed. The test sample shall be rinsed with water and dried. After drying, the area of the coating subjected to the test shall be examined in accordance with the following method:

a) Inspect and assess each coating immediately after the test period. Detach the plastic pipe from the test site. Using a lint-free paper towel, wipe along the surface of the coating and cathode area to remove moisture.
b) Make about 12 radial incisions using a sharp knife through the coating to the substrate extending outwards from the holiday for a distance of at least 40 mm. Make these incisions at an angle of approximately 30° from each other.
c) Insert the knife point into the centre portion of the holiday down to the metal substrate. Using a gentle levering action, peel away slowly a radial section of coating continuing until firm adhesion is encountered.
d) As loss of adhesion is not always obvious, carefully examine the substrate for sign of residual coatings which indicates that disbonding has not occurred.
e) Repeat with each radial segment.

H-6 RESULTS

The result of the length of the cathodic disbondment is defined as the arithmetic mean value of the 12 single values. The mean value shall be reported.

ANNEX J

(*Clauses* 10 *and* 13)

FLEXIBILITY TEST

J-1 GENERAL

This procedure shall be used to assess the flexibility of three layer polyolefin coatings, applied to a bare steel substrate.

J-2 EQUIPMENT

The equipment shall consist of the following:

a) Hydraulic press;
b) Bending mandrels with fixes radii;
c) Freezer; and
d) Strain gauges, if applicable.

J-3 SPECIMEN

Coated test samples shall be cold cut from a pipe ring with a minimum length of 200 mm parallel to the axis of the pipe. The width shall be approximately 25 mm.

J-4 PROCEDURE

Smooth the coating on the edge of the test sample to remove any potential stress risers. Place the test specimen in the freezer, cool it to within 3°C of the powder manufacturer's certified minimum flexibility test temperature of 0°C, and hold it within that temperature range for a minimum of 1 h.

Determine the effective sample thickness t which includes the actual sample thickness and any curvature, by placing the specimen on a flat surface and measuring the effective thickness shown in Fig. 8.

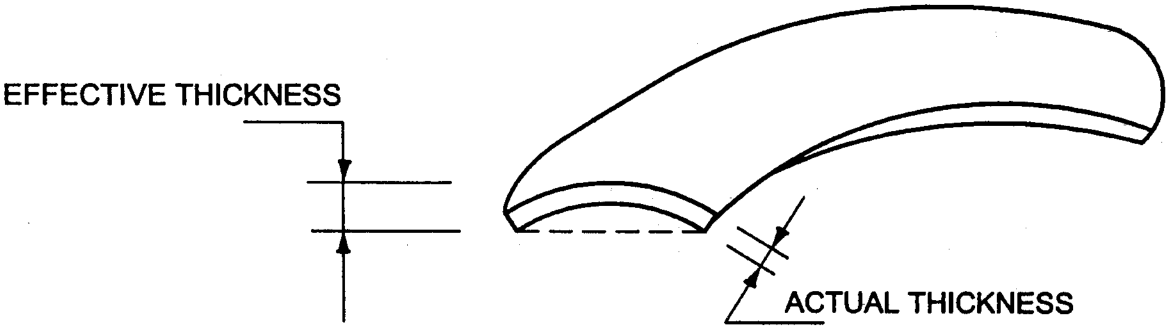


FIG. 8 EFFECTIVE THICKNESS

Determine the mandrel radius that corresponds to an angle of deflection of 2° per pipe diameter length by using following formula:

$$R = 28.15\,t \qquad \ldots 1$$

where

R = mandrel radius in mm, and

t = effective sample thickness, in mm.

Bend the test sample over a mandrel whose radius is not larger than that determined in accordance with equation 1. Bend the specimen such that the operation lasts no longer than 10 s and is completed within 30 s of the test samples having been removed from the freezer.

NOTE — If the specimen exhibits peaking the percent strain can be calculated by the use of strain gauges attached to the test sample.

J-5 TEST RESULTS

Warm the bent test sample to 20 ± 5°C, and hold it in this temperature range for a minimum of 2 h. Within the next hour, visually inspect it for the presence of cracks.

ANNEX K

(*Clauses* 10 *and* 13)

HOT WATER SOAK TEST

K-1 GENERAL

This procedure shall be used to assess the comparative resistance of three layer polyolefin coatings, applied to a bare steel substrate, to loss of adhesion due to hot water immersion.

K-2 EQUIPMENT

The following equipment shall be required:

a) oven, or hot water bath, controllable to within 3°C;

b) holiday detector; and

c) pointed sharp knife, that is, stanley type or similar.

K-3 SAMPLE PREPARATION

Cold cut 150 mm pipe rings with the factory applied coating and joint coating. If the pipe diameter is too large, 100 mm × 100 mm × wall thickness cold cut panels may be utilised.

When cold cutting operations are being undertaken care shall be taken to ensure that the cutting operation does not 'rip' the coating.

To ensure freedom from holidays, the coated pipe samples or rings shall be tested for holidays with the holiday detector set at the required voltage for the particular coating system.

K-4 TEST PROCEDURE

K-4.1 Duplicate samples per coating shall be used for evaluation for procedure qualification testing, and for production testing.

The steel and the steel/coating interface shall not be protected.

K-4.2 Visually assess the initial adhesion on all samples to make sure that the full coating system is adherent to the substrate.

K-4.3 Insert the test samples in a water bath containing tap (potable) water, heated to 65°C.

K-4.4 For procedure qualification trials remove duplicate samples after 24 h, allow to cool to ambient temperature and assess the adhesion of the edge of the coating to the substrate.

K-5 ADHESION TEST PROCEDURE

For all 3-layer polyolefin coatings a visual assessment of all test samples shall be undertaken along the total interface of the coating with the substrate.

K-6 REPORT

Record any loss of adhesion of the coating to the substrate, and/or any loss of adhesion between the layers.

ANNEX L

(*Clause* 8.3)

GEL TIME OF THE EPOXY POWDER

L-1 GENERAL

This procedure shall be used to assess the gel time of the epoxy powder used in the three layer coating.

L-2 EQUIPMENT

The equipment shall consist of the following:

- a) hotplate controllable to within 3°C;
- b) metal plate placed on top of the hotplate;
- c) stopwatch or electric timing device capable of measuring 0.1 s intervals; and
- d) draw down tool (*see* Fig. 8).

L-3 PROCEDURE

L-3.1 Conduct three tests and average the results.

L-3.2 Heat and maintain the temperature of the metal plate surface that will be in contact with the powder at a temperature of 205 ± 3°C.

L-3.3 Cover the bottom 25 mm of the draw down tool with epoxy powder.

L-3.4 In a smooth motion, deposit and draw the epoxy powder across the metal plate while holding the tool at an angle of approximately 45° to the metal plate, thereby creating a tongue of epoxy powder approximately 25 mm wide.

NOTE — The target thickness of the cured film is 300 µm to 400 µm.

L-3.5 Start the timing device and deposition of epoxy powder on the metal plate surface simultaneously.

L-3.6 Applying light pressure on the draw down tool, repeatedly draw the edge of the tool through the melted epoxy powder. Stop the timing device when the tool rides up on the gelled epoxy powder and no longer contacts the metal plate.

L-4 RESULTS

The following information shall be reported:

- a) Epoxy powder batch number;
- b) Date of testing; and
- c) Gel time, in seconds.

ANNEX M

(*Foreword*)

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